UTAH DEPARTMENT of ENVIRONMENTAL QUALITY
WATER QUALITY

Sampling and Analysis Plan: Utah Lake Littoral Sediment Study: An Assessment of Carbon, Nitrogen, and Phosphorus Dynamics in the Utah Lake Littoral Zone

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Prepared by the Littoral Sediment Team

List of Contributors

PI: Erin Rivers, Utah State University
Co-PI: Dr. Zachary Aanderud, Western States Water and Soil & Brigham Young University
Co-PI: Dr. Greg Carling, Western States Water and Soil & Brigham Young University

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Acronyms/Abbreviations

USU	Utah State University
BYU	Brigham Young University
DWQ	Division of Water Quality
CUWCD	Central Utah Water Conservancy District
ULWQS	Utah Lake Water Quality Study
SAP	Sampling and Analysis Plan
P	Phosphorus
N	Nitrogen
PAR	Photosynthetically active radiation
SOP	Standard operating procedures
DIN	Dissolved inorganic nitrogen
SRP	Soluble reactive phosphorus or orthophosphate
TN	Total nitrogen (mg/L)
TP	Total phosphorus (mg/L)
DEA	Denitrification enzyme activity

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1. Introduction

With the large variations in lake levels and shallow depth, large expanses of Utah Lake's littoral sediments are subject to wetting and drying cycles of varying durations and frequencies. As littoral sediments go through periods of desiccation and inundation, it changes sediment properties and alters the duration of oxic and anoxic conditions, which in turn affects sediment oxygen demand, C, N and P release as well as microbial activity and composition (Weise et al., 2016). A number of studies have found that sediment drying promotes the release of potentially significant amounts of bio-available N and P on re-wetting (the so-called "Birch effect"; Baldwin and Mitchell, 2000; Birch, 1960; McComb and Qiu, 1998; Scholz et al., 2002). This occurs as a result of numerous interacting processes, including enhanced aerobic microbial mineralization of organic matter (OM) and the reduction of nitrate, leading to an accumulation of ammonium N in the sediment; a decreased capacity of the sediments to adsorb nutrients such as P (Baldwin, 1996); and the release of cell-bound nitrogen (ammonium) and filterable reactive phosphorus from sediment bacteria as they are killed during drying (Qiu and McComb, 1995). Although both N and P may be released by these processes, they may respond differently, since the re-wetted sediments may have a reduced capacity to release P under anoxic conditions (which suggests that more N than P could be released into the water column on lake filling) (Mitchell and Baldwin, 1998). The degree and duration of drying before rewetting has been shown to affect nutrient release. Schönbrunner et al. (2012) performed an internal phosphorus loading study in which floodplain sediments were exposed to different dry/wet treatments. They found that total phosphorus (TP) release from sediments into the water column increased with increasing duration of dry periods prior to rewetting and that repeated drying and wetting resulted in elevated phosphorus release. This effect was more pronounced when drying periods led to an 80% reduction in water content.

Sediment characteristics also affect nutrient releases. Shaughnessy et al. (2019) found that spatial distributions of lakebed nutrients in an agricultural reservoir in Illinois were predominantly controlled by sediment depositional patterns. The largest proportion of clay-sized particles and highest concentrations of OM were deposited near the dam wall and the highest proportion of (heavier) sand-sized particles were deposited near the river mouth. They found a significant and positive correlation between TP, TN, and TC with OM. Shaughnessy et al. (2019) also found that seasonal factors were important to consider. Nitrogen species varied seasonally at the sediment-water interface and were significantly higher during warmer weather/the growing season. The warmer conditions may enhance the release of nutrients from the sediments to the water column due to higher decomposition rates, higher pH due to photosynthetic activities, and low DO near the sediment-water interface that can change redox conditions so that reduced iron (Fe) might liberate P.

Little is currently known about the effects of water level fluctuations/wet and dry phases on C, N, and P loading from littoral sediments in Utah Lake. As the Science Panel works to respond to charge questions and nutrient criteria are being developed, it is important that this knowledge gap be addressed. More specifically, the Science Panel needs to better understand whether littoral sediments act as nutrient sinks (e.g., through denitrification, respiration and

sedimentation) or sources (e.g., decomposition/mineralization and release upon rewetting), to what magnitude, and whether they reduce or enhance nutrient loads and impact the overall nutrient budget of the lake. The Science Panel also needs quantitative relationships between the duration and frequency on wetting and drying on nutrient loading in order to evaluate relationships between external and internal loads to Utah Lake. A comprehensive understanding of the influence of fluctuating hydrological conditions on internal nutrient loading within Utah Lake is critical in developing restoration and management plans, and this study will inform these solutions.

2. Background

The Utah Department of Environmental Quality, Division of Water Quality (DWQ) is contracting with Utah State University to conduct a littoral sediment study to help understand effects of drying/wetting on Carbon (C), Nitrogen (N) and Phosphorus (P) flux from littoral sediments in Utah Lake. This study was prioritized by the Utah Lake Water Quality Study (ULWQS) Science Panel. The target completion date of this scope is April 30, 2022.

The Utah Division of Water Quality (DWQ) is in Phase 2 of the Utah Lake Water Quality Study (ULWQS) to evaluate the effect of excess nutrients on the lake's recreational, aquatic life, and agricultural designated uses and to develop site-specific nitrogen and phosphorus water quality criteria to protect these uses. The ULWQS is guided by the Stakeholder Process developed during Phase 1, which established a 16-member interest-based Steering Committee and a 10-member disciplinary-based Science Panel. The Steering Committee charged the Science Panel with developing and answering key questions to characterize historic, current, and future nutrient conditions in Utah Lake. Responses to the key questions will be used by the Steering Committee to establish management goals for the lake and by the Science Panel to guide development of nutrient criteria to support those goals.

Water level data have been collected in Utah Lake since the late 1800's (Figure 1; CUWCD and Thurin 2007). A probability distribution of fluctuations in lake area using data from 2004-2018

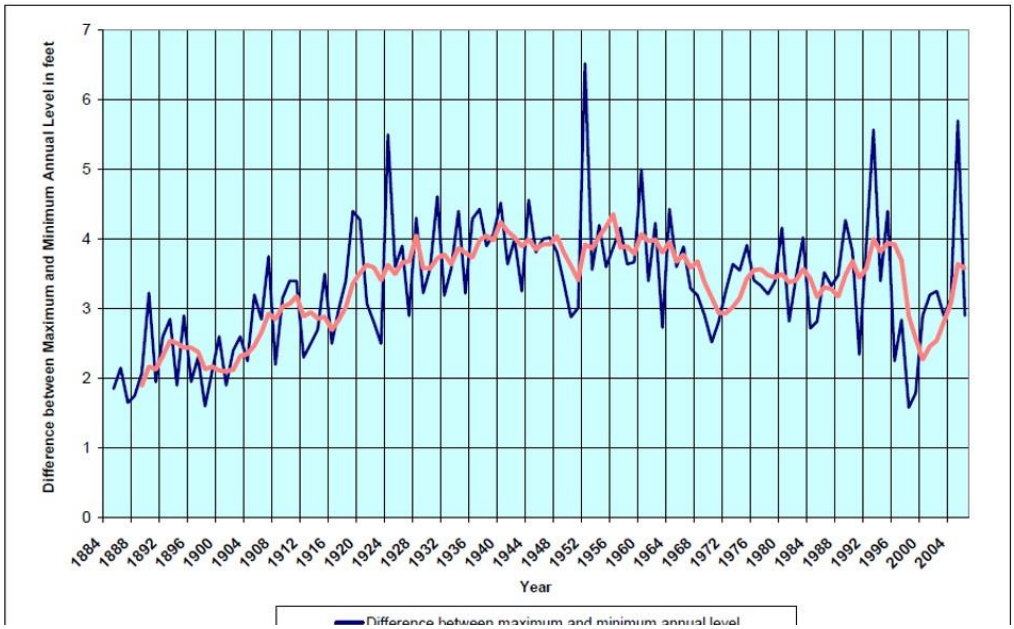

Figure 1. Annual and five-year average within-year variation in Utah Lake level from 1884 to 2006 showing generally increasing variation (doubling) over the historical period from 1884 to the 1930s to 1940 (from CUWCD and Thurin 2007; Figure 11).

estimated that the 5 to 95th percentile in lake area varied by 30 mi^2 from an average of 130 mi^2 (J. Martin, pers. comm).

Based on other estimates, approximately 10-15% of the area is littoral. The extent of the areas of wetting and drying can also be illustrated by the Utah Lake bathymetry (Figure 2) comparing areas that were always wet to those that were periodically dry. In either case, the amount of lake area potentially affected by wetting and drying is substantial. The duration of dry and wet phases can also be inferred from lake level data and can range from months to years.

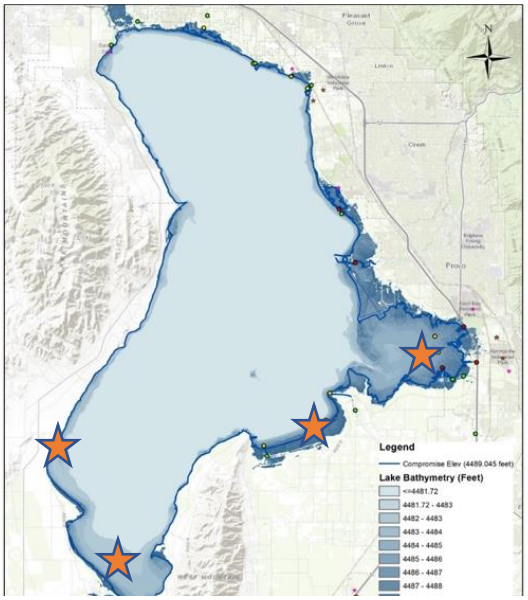

Figure 2. Areas of Utah Lake that were continuously wet for the period of 2010 – 2020 (light blue) versus those that were periodically dry (dark blue bands).

Other existing, complementary studies include a project recently completed by Goel and Carling on sediment–water-nutrient interactions in Utah Lake[1] (Goel et al., 2020). Results include calculations of sediment fluxes over a range of water column P concentrations and an exploration of the potential

effects of changing pH, alkalinity, and redox. Equilibrium P concentration, the water column concentration at which the sediment switches from a sink to a source of P, were estimated from this study as well. The study also estimated sediment oxygen demand and provided information on the role of sediment resuspension on nutrient releases or removal, primarily via calcite scavenging. The experiments were performed on wet cores collected from two sites —one site in Provo Bay and one site in the main body of the lake at an established DWQ monitoring site. These data could be contrasted with the results from this work, but also provide information on wet core nutrient content and flux rates. Aside from that, little is known about nutrient release from littoral sediments in Utah Lake, thus the need for this work.

2.1. Problem Statement

There is a high degree of annual variability in water levels in Utah Lake, resulting from a combination of natural and anthropogenic factors. Variability in precipitation patterns, evaporation, upstream water use, and managed outflow contribute to the hydrologic fluctuations that result in 3-4 feet variability in lake levels throughout the year. Due to the shallow nature of the lake, fluctuations in water levels cause major changes in water-edge location and lake characteristics. Currently, little is known about the effects of dry and wet phases resulting from water level fluctuations in Utah Lake.

2.2. Study Objectives

The objective of this research is to address the following question identified by the ULWQS as critical to understanding the current state of Utah Lake with respect to nutrients and littoral sediment. More specifically, this research will help inform the following charge questions:

- If there are linkages between changes in nutrient regime and Harmful Algal Blooms (HABs), what role if any does lake elevation change play? (Science Panel charge 2.3.iii)
- What is the sediment oxygen demand of, and nutrient releases from, sediments in Utah Lake under current conditions? (Science Panel charge 2.4.ii)

The study is designed to address the following tasks:

1. Task 1 – Frequency and Duration of Sediment Drying-Rewetting on Nutrient Release and Oxygen Demand

   Quantify the relationships (for Utah Lake) between the frequency and duration of dry periods on the subsequent nutrient releases and oxygen demand following re-wetting through field and laboratory studies.

2. Task 2 – Rate and Magnitude of C, N, and P Fluxes from Drying-Rewetting Sediments

Quantify the rate and magnitude of nutrient (C, N, and P) fluxes following drying and re-wetting over a range of sediment characteristics and wetting/drying phases from littoral sediments.

3. Task 3 – Spatial and Temporal Model on Impact of Drying-Rewetting Sediments

   Determine the spatial and temporal extent (duration and frequency) of wetting and drying patterns in littoral areas through Geographic Information System (GIS) analysis and evaluation of daily lake elevation data. Develop quantitative relationships for estimating the oxygen demand and nutrient fluxes of re-wetted sediments as a function of the frequency and duration of periods of wetting and drying. These objectives will be completed by DWQ in consultation with the Littoral Sediment Team.

Task 1 will allow for determination of the mass fluxes of C, N, and P associated with wetting and drying in Utah Lake's littoral zone, while Task 2 will provide valuable mechanistic insights into how microbial activity at the sediment-water interface may influence the overall mass fluxes being measured, and the potential reactivity of N being loaded into the water column upon inundation. Such mechanistic insights are valuable both for upscaling our mass flux measurements to the full lake area and may additionally provide management implications for ways to reduce C, N, and P fluxes from the sediments. USU Research Team will work directly with the Division of Water Quality to upscale our measurements to the full lake area in Task 3.

2.3. Expected Outcomes and Deliverables

Specific outputs are expected to include, but are not limited to, a summary of existing literature and data, anecdotal information on the effects of drying and wetting on lake littoral sediment nutrient flux, a sampling and analysis plan (SAP), the project dataset, and a technical report with detailed results for all tasks. All data collected for this project will be made available to the Science Panel per the deliverable dates schedule in Section **Error! Reference source not found.**.

The littoral sediment SAP describes field, laboratory, and experimental techniques, data quality objectives, and analytical approaches employed for this study. The SAP prioritizes well-established, applicable techniques, over development of exploratory or experimental techniques. The SAP is developed in accordance with the DWQ's *Quality Assurance Program Plan for Environmental Data Operations, Final Plan* (Revision No. 1.0) and describes the ten essential elements listed in Appendix A. The SAP document must receive Science Panel approval prior to initiation of sample collection and experimentation (Task 3) to ensure this research will accomplish the stated study objectives.

When this study is completed, the Science Panel will be able to answer the study objectives listed above and understand, with greater certainty:

- The role of littoral sediments subject to wetting and drying as sinks and/or sources of C, N, and P.
- The amount of nutrient loads from those areas of the littoral zone subject to wetting and drying relative to other internal and external loads.
- The effects of varying patterns of drying/wetting (e.g., duration and extent) on C, N, and P flux from littoral sediments.

To characterize sediment C, N, and P dynamics in response to hydrologic shifts in littoral zones with fluctuation lake levels, we will carry out two sets of experimental tasks to determine 1) the effects of frequency and duration of sediment drying and rewetting periods on net nutrient release and oxygen demand and 2) the mechanisms regulating the release of C, N, and P in response to hydrologic fluctuations.

3. Task 1 – Frequency and Duration of Sediment Drying-Rewetting on Nutrient Release and Oxygen Demand

3.1 Study Design

To determine N, and P fluxes from drying-rewetting sediments, we will perform experiments using intact sediment cores collected along transects in the Utah Lake littoral zone. To allow the sediments to dry in a timely fashion and to capture the N and P release rates with the most interactive sediment-water interface, we will restrict cores to contain approximately 10 cm of sediment. At four lake sites, detailed below, we will extract cores from three locations experiencing different lake water fluctuations and frequencies of drying and re-inundation: lake location–in the lake that does not experience drying and re-inundation, lake margin location–the water margin that may experience periodic drying and re-inundation that is currently under water at the edge of the lake surface, and exposed lake sediment location–sediment that is now exposed but as the lake level rises may be re-inundated with water in the future. At each of the four lake sites and the three drying and re-inundation locations, we extract two sets of cores that will be experimentally manipulated. The first set of cores will be continually wetted or flooded for the duration of the experiment (**control cores**) to simulate an absence of drying and re-inundation. The other set of cores will experience a dried and re-inundate (**drying and re-inundation cores**). The core tubes are composed of clear acrylic plastic with dimensions of 50 cm length × 5 cm diameter.

For the entire experiment we will have four lake sites × three drying and re-inundation locations × two experiment manipulations (control, drying and re-inundation cores) × three replicates × nine repeated nutrient evaluation measurements through time for a total of 72 cores and 648 total samples for nutrient analyses. We will also analyze the nutrient concentrations in sediments and sediment chemistry on 12 cores (four sites × three locations).

The four lake sites will cover a range of nutrient concentrations across Utah Lake to provide a weighted average of sediment nutrient conditions across the littoral zone. Based on a map of sediment P concentrations from a published study (Randall et al., 2019) and a map of the littoral zone in the lake (Figure 2 of this SAP), we have selected the following sites to collect sediment

cores: 1. Provo Bay (high P concentrations; 2. Goshen Bay (moderate P concentrations); Spanish Fork River delta (moderate P concentrations); and 3. Southwest shoreline (low P concentrations). The sites are shown as gold stars Figure 2.

3.1.1 Sediment Drying and Re-inundation Experiment

The drying and re-inundation experiment will consist of two drying and rewetting periods. The experimental drying and re-inundation manipulation will go as follows:

1. We will remove all the residual water from the cores and immediately add 580 mL of filtered lake water to the control cores.

- Water will be removed and added carefully to prevent disturbing the sediment surface. Water for the experiments will be collected at the same lake location as the sediment cores at each lake site. Thus, the water in each core will be site-specific and contain the bacteria and lake chemistry local to the site. The volume of water added to each core will make a water column height of ~30 cm above the sediment. Because sediment cores often produce extra nutrient fluxes after disturbance during core collection, at least three additional cores will be collected at one location and left to stabilize for one week prior to removing and adding new lake water. We will compare nutrient fluxes from the stabilized cores with other control cores to determine potential differences in flux rates.
- When water is present in the cores the cores will be capped to prevent evaporation via a small hole in the cap to allow gas exchange.
- We will bubble air/oxygen into the lake water of all 72 cores to maintain the oxygen supersaturated conditions common in Utah Lake. Filtered air will be bubbled into cores with aquarium bubblers and any particulate particles in the air will be removed with an 0.45 μm inline filter.
- All cores will be wrapped in tinfoil to discourage cyanobacteria and total algae growth that may foul the tubes and act as a significant sink for N and P being released. The manipulation will still allow bacteria in the water column to utilize the N and P being released from the sediments.

2. We will allow the drying and re-inundated cores to dry down for two to three weeks at room temperature until they reach a constant mass. We expect the sediments to become oxic over time and bacteria to become inactive.

- The cores that are being dried will be uncapped and covered with gauze to help with sediment drying and prevent foreign bacteria and fungi from entering the cores.
- The drying and re-inundated cores will be weighed every other day until they reach a constant mass.

3. We will add 580 mL of lake water to the drying and re-inundated cores, following the same procedures outlined in step 1 for control cores.

4. When the cores are wet, we will analyze water chemistry on control and re-inundated cores periodically over a three-week rewetting period, specifically at days 1, 5, and 21.

- Since the control core will be re-inundated two to three weeks before the re-inundated cores, the nutrient analyses for the cores will be offset by a few weeks.
- We will replace the sampled water volume removed for analyses with filtered lake water.
- We will analyze the nutrient concentrations in the stored site water so we will be able to calculate the N and P concentrations added to the cores.

5. We will remove water from the drying and re-inundated cores and allow the cores to dry down again for two to three weeks to a constant mass. We will then add another volume of water to the core (step 1) and sample the water over a three-week wetting period (step 4).

6. After the second round of drying and re-inundation, we will perform a P binding experiment to quantify P uptake in the sediments. We will add orthophosphate at a concentration of 10 mg-P L^{-1} as K_2HPO_4 to saturate the water column with available P. We will dry down the cores again for two to three weeks to a constant mass and add the P in the water during re-inundation. The same amount of P will be added to the control cores. We will measure the changes in water chemistry three times over a three-week flooding period as described in step 4. The amount of N and P sequestered by the sediment will be estimated by the loss of N and P in the water column.

7. After we are finished with nutrient sampling from the cores, we will estimate sediment oxygen demand by turning off the air bubblers to allow the sediment cores to go anoxic. We will measure dissolved oxygen concentrations in the cores hourly using a handheld DO meter. The DO response will be compared for the control cores and the wetting/re-inundation cores to determine impacts of wetting and drying on sediment oxygen demand.

3.2 Sampling Supplies

3.3.1 Field supplies

- ☐ Boat
- ☐ Keys
- ☐ Full tank of gas
- ☐ Anchors
- ☐ Life jackets
- ☐ Percussion corer with extension poles
- ☐ 84 plexiglass core sleeves (50 cm × 5 cm)
- ☐ 84 core catchers
- ☐ 168 caps
- ☐ Tape measure for measuring core depth
- ☐ Electrical tape
- ☐ Aluminum foil
- ☐ Cooler with ice, retrofitted to hold cores upright, covered with black plastic bag
- ☐ Shovel
- ☐ Disposable gloves
- ☐ Four buckets
- ☐ 80 one-gallon jugs (20 gallons of lake water per site)
- ☐ Cooler with ice for water samples

- Three paddles
- Wisconsin net to filter out zooplankton
- EXO, cable, and handheld
- GPS
- Field notebook with pencil
- Paper towels
- Wash cloths
- Camera/phone
- Sharpies
- DI

3.3.2 Lab supplies

- PVC racks to hold cores
- Vacuum filtration unit with pump
- 80- l gallon jugs for storing filtered water
- Peristaltic pump for removing overlying water from cores
- Tubing to carefully transfer filtered water back into cores
- Core aeration: 72 aeration stones, aquarium pumps, tubing

3.3.3 Sampling water N and P

- 72 syringes for periodic water samples from each core
- 648-0.45 µm nylon syringe filters
- 648-50 mL sample vials for unacidified water samples
- 648-50 mL sample vials for acidified water samples
- HNO3 to acidify water samples for ICP-OES analyses
- 648-30 mL glass vials for DOC samples
- 648 GFF filters for DOC
- 648 alkalinity test kits for DIC

3.4 Sampling Measurements and Analyses

3.4.1 Water Chemistry Analyses

In-situ physicochemical analyses will be conducted with a YSI water chemistry sonde (Yellow Springs Instrumentation, USA) in the lake waters and in the cores at each sampling time. The sonde will estimate pH, electrical conductivity, and dissolved oxygen in the water column (Jones et al., 2017).

3.4.2 Water N and P Analyses During Drying and Re-inundation

The list of nutrient analyses in the water column are as follows with attending hyperlinks for SOPs within the Research Current Utah Lake Littoral Folder/Littoral Sediment SOPs (email Zach Aanderud for access permission to box folder containing SOPs):

- Total N (TN) / Total P (TP) https://byu.box.com/s/gpxbiw0t6rroldfba8c1s468b7h5x3zj
- NH_4^+-N / NO_3^--N https://byu.box.com/s/tv1xi46uzmjlwq6owgttp4ikxoytrqox
- Soluble reactive P (SRP) https://byu.box.com/s/esjvwkdr5dsiu0mti163r2woknqc2qbc
- Total dissolved P (TDP) same method as TP but on fileted samples through a 0.40 μm filter
- Particulate P (PP) using the formula TP – TDP
- Dissolved organic C (DOC) see below
- Dissolved inorganic C (DIC) see below

All water chemistry analyses will be evaluated using standard methods at the Environmental Analytical Laboratory (EAL) at BYU. We analyzed four forms of P and three forms of N to evaluate nutrient release from sediments. The five forms of P vary in reactivity and potential bacterial use, with the forms including and TP, a conglomerate measurement of P in both organic and inorganic forms; TDP, a conglomerate measurement of dissolved organic and inorganic P; PP, P bound to colloids or assimilated within other organisms and presumed to be relatively unavailable to bacteria; and SRP, an inorganic form of P, mostly orthophosphate, that is the most bioavailable to bacteria. Briefly, we analyzed: TP concentrations (unfiltered sample) by using a nitric acid microwave assisted digestion followed by determination with a Thermo Scientific ICP-OES (iCAP 7400, Thermo Electron, Madison, WI, USA); SRP with the ascorbic acid method (4500-P.F.) (Koenig et al., 2014); and TDP (filtered sample though a 0.40 μm filter) on a Thermo Scientific ICP-OES (iCAP 7400, Thermo Electron, Madison, WI, USA). We calculated PP using the formula TP – TDP. DOC will be measured on a TOC-L series total C analyzer following standard methods on a Shimadzu TOC analyzer (TOC/TN-L, Shimadzu Scientific, Kyoto, Kyoto Prefecture, Japan). and DIC will be measured by acid titration using HACH Alkalinity test kits. The detection limits for all three N forms are as follows: NH_4^+-N = 0.03 mg/L / NO_3^-N = 0.02 mg/L, and TN = 0.02 mg/L. The limit of detection for all P forms is < 0.005 mg/L.

3.4.3 Sediment and Sediment Chemistry Analyses

To characterize differences in sediment nutrient, chemical, and physical properties across our lake sites to determine the amount of N and P present in the cores at the beginning of the experiment and the sediment chemistry to explain potential differences between release N and P. One extra core from each of the site by lake location (12 total) will undergo a suite of analyses. We will homogenize the 10 cm of sediment from each core and measure sediment density, water content after oven drying, and organic matter content by loss on ignition. We will also measure TC, TN (combustion), TP (ICP-OES), and metals (ICP-OES) using similar protocols as described above for water samples. We will estimate P speciation by sequential extractions following a protocol optimized for use in calcite-rich lake sediments (Hupfer et al., 2009). The extract steps are analyzed for TP and/or SRP following procedures described above for water samples. Sediment mineralogy will be analyzed using a Rigaku MiniFlex Benchtop X-Ray Diffractometer (Rigaku, Tokyo, Japan).

3.5 Sample Transport and Storage

All cores will be transported to the lab on ice prior to starting the drying-rewetting regimes. The drying and re-inundation events will be initiated within 12 hours of core extraction and manipulations will be conducted at room temperature 20°C.

3.6 Task Milestones and Deliverables

The data from the drying and re-inundation experiment will provide an overall approximation of the N and P release rates during the re-inundation of dry sediments. Utah Lake experiences lake water fluctuations that cause sediments to dry and become re-inundated. Generally, Utah Lake dry littoral sediments may experience one re-inundation event as the lake level rises in the spring and declines in the fall or sediments that straddle the margin of the lake may experience multiple intra-annual fluctuations and drying-re-inundation event. Further, Utah Lake levels may also remain low over multiple years, leading to the re-inundation event of sediments that were previously saturated or periodically exposed to drying. Our experiments including the three lake location (i.e., lake location, lake margin location, and sediment location) will simulate all three of these drying and re-inundation regimes. We will also estimate the mineral sink potential of the sediments for P.

We will calculate the release rates or fluxes between sediment and the water column with the following equation:

$$\text{Nutrient flux (mg m}^{-2}\text{ day}^{-1}\text{)} = dCe/dt \times V/A \times 1000\text{ mg g}^{-1} \times \text{day}^{-1} \quad \text{(equation 1)}$$

where, dCe = change in nutrient concentrations in the water column (mg L^{-1} = g m^{-3})
dCe/dt = change in nutrient concentrations over time (g m^{-3} day^{-1})
V = volume of overlying water in the core (m^3)
A = sediment surface area in the core (m^2).

The release the release rates of N and P across the sediment-water interface will be used to inform Task 3 and describe the potential nutrient loading of sediments from under different drying and re-inundation conditions.

4. Task 2 – Rate and Magnitude of C, N, and P Fluxes from Drying, Dry, and Rewetting Sediments

4.1 Study Design

To determine mechanisms of C, N, and P release from sediments with various hydrologic histories under dry and wet conditions and undergoing wet-to-dry and dry-to-wet transitions, we will examine the rate and magnitude of several biogeochemical processes that regulate the production and consumption of C, N, and P under various hydrologic conditions. We will evaluate sediment metabolic function under experimental drying and inundation scenarios and measure *in situ* fluxes of C and N gases.

For this experimental task, Rivers and students will carry out four field sampling campaigns, during which lake sediment C and N fluxes will be assessed during dry summer conditions and transitional fall conditions. This part of the project will not experimentally manipulate moisture content but will assess *in situ* conditions during a dry to wet period to understand the mechanistic partitioning of production and consumption processes under variable hydrologic conditions. In this phase, C and N fluxes will be measured along a dry to wet gradient of *in situ* Utah Lake sediments in 2 parts:

1. Sediment cores will be collected along the same dry to wet gradient of *in situ* Utah lake sediments as in Task 1 (inundated lakebed, intermittent littoral, and perennially dry sediment locations) within the 4 transects around the lake. Project steps will include four sampling events at Utah Lake from August to December 2021. Four cores will be extracted at each sampling location; two cores will be capped and remain intact, and two will be composited and homogenized for subsampling for N and P analyses, sediment composition, organic matter content, porosity, bulk density, and C/N/P content. If water is present, three water samples will be collected and filtered on site. Samples will be put on ice and transported back to the laboratory for analysis. Sediment surface aquatic metabolism (gross primary production, ecosystem respiration, net ecosystem production) rates and sediment nitrogen cycling (denitrification, net nitrification, net mineralization, N release from microbial biomass) will be measured in sediments collected from each lake sampling location. Methods are detailed in sections 4.1.1. and 4.1.2.
2. *In situ* C and N flux will be measured at exposed, dry sites using greenhouse gas chamber experiments.

4.1.1. Carbon: Benthic Metabolism

Sediment surface aquatic metabolism (gross primary production, ecosystem respiration, net ecosystem production) rates will be quantified by manipulating sediment cores retrieved from the lake. The overarching goal of doing this is to create a production-irradiance (P-I) curve (Jassby and Platt, 1976) so that benthic primary production can be modeled given known light conditions in a lake (Brothers et al., 2016; Vadeboncoeur et al., 2008). A P-I curve provides the initial response of primary production to photosynthetically-active radiation (PAR) (α), the maximum light-saturated rate of photosynthesis (P_{max}), and the PAR at which P_{max} is achieved (I_k).

When illuminated, both oxygen consumption (ecosystem respiration; ER) and oxygen production (gross primary production; GPP) are occurring simultaneously. The net change in oxygen under illuminated conditions is thus called net ecosystem production (NEP). Under dark conditions, only ER is occurring, as photosynthesis is halted. GPP can thus be calculated as the difference between the rates of change in dissolved oxygen between light (NEP) and dark (ER) incubations (i.e., GPP = NEP – ER). Although there are other non-oxygenic ways to measure aquatic metabolism, dissolved oxygen is the most widely used approach (Staehr et al., 2012).

Algal light adaptation and production partitioning between the sediments and water column need to be accounted for in measurements. Regarding the former – primary producers can adapt to low and/or high-light conditions, so standard procedure for determining algal responses to light supply are to dark-adapt algal communities for at least 15 minutes prior to exposing them to a given light intensity. Regarding the latter – a retrieved core containing sediments and lake water will have algae suspended in the water column (phytoplankton) as well as algae growing on the sediment surface (periphyton). Lake water will be carefully poured out (minimizing disturbance of the biofilm community at the sediment-water interface), filtered to remove phytoplankton (0.45 μm), and returned to the core tube to ensure that only periphyton production is being measured during the experiments. As periphyton will also likely grow on the in-facing side of the plastic cores, we will thoroughly clean that surface with a brush before beginning experiments.

The oxygen gradient at the sediment-water interface can be extremely steep (i.e., within millimeters), even when the waters are being physically stirred (Carlton and Wetzel, 1987; Vadeboncoeur and Lodge, 1998) and this can be a potentially large source of error. Therefore, we will use a micro-O_2 sensor (PreSens) to measure oxygen dynamics at the sediment-water interface.

These experiments will be carried out according to the following steps:

1) Cores will be collected from lake sampling locations using a coring tube with appropriate dimensions for the oxygen-measuring lab equipment. The top and bottom of core will be plugged with rubber stoppers, and extra water will be collected for top-up in lab. Sediment-to-water ratio will be measured and recorded, ensuring that enough sediments should be present to provide for a stable sediment-water interface for lab measurements (i.e. > 5 cm).
2) Cores will be transported upright to the laboratory, minimizing disturbance to the sediment-water interface. Cores will be stored at ambient room temperatures, approximating ambient summertime Utah Lake temperatures.
3) Water will be drained from cores, filtered, and returned to the cores for measurement of sediment metabolism dynamics. Any algal growth will be washed out prior to water transfer.
4) Cores will be arranged under LED grow lights.
5) Starting with a dark incubation measurement, sampling time, water temperature, and dissolved oxygen concentration (mg/L) are recorded. The dark incubation period will be 30 minutes with measurements every 5-10 minutes.
6) LED lights will then be turned on and paired with a maximal amount of shading cloth between the sediments and the lights (i.e., the lowest possible direct light intensity affecting the sediment surface). Using a light meter, we will measure the light intensity reaching the sediments. The same sampling intervals will be used as in step 5, but in this case the slope in changing DO will be the net ecosystem production (NEP).
7) The core will be returned to a dark incubation period after each light period (i.e., alternating light-dark incubations), with each light incubation being incrementally higher light intensity than the previous one. This approach provides a large number of ER rates

that can be averaged for an overall mean ER, it acts to dark-adapt each light incubation, minimizing super- or under-saturation of DO in the water column.

4.1.2. Benthic Nitrogen Cycling

We will evaluate how rewetting events that cause sediments to go from dry to flooded conditions affects dissolved N pulses that may lead to significant release of reactive N from the sediment microbial biomass. As N fluxes may play an important role in Utah Lake productivity, and N cycling dynamics are more complex than those of P, we plan to further assess detailed N dynamics with inundation. The following analyses will provide a better understanding of the potential for N removal via denitrification, and microbial contributions to reactive N.

Homogenized sediment samples at each lake location will be subsampled for analysis of ambient denitrification rates, denitrification enzyme activity, net mineralization rates, net nitrification rates, and microbial biomass N. Ambient denitrification rates and denitrification enzyme activity (DEA) will be analyzed to estimate *in situ* sediment denitrification rates and determine potential sediment denitrification rates, respectively. The chloramphenicol-amended acetylene-block method (Smith and Tiedje, 1979). Triplicate soil slurries of 10 g sediment and 10 mL lake water will be added to 125 mL glass flasks capped with septa. The DEA set will be amended with NO_3 (as KNO_3) and organic carbon (as dextrose), and the ambient denitrification set will not receive any amendments. We will add chloramphenicol, an antibiotic that inhibits the production of new enzymes, allowing for denitrification rates measured in bottle assays to be more representative of denitrification activity at the time of sampling (Smith and Tiedje, 1979). The flasks will be purged with helium to remove oxygen and force anaerobiosis. We will inject 5 mL of acetylene gas into the sealed, anoxic microcosms through septa caps using a syringe. Acetylene inhibits the conversion of nitrous oxide (N_2O) to dinitrogen (N_2) by blocking the activity of nitrous oxide reductase, allowing the measurement of N_2O accumulation to estimate denitrification rates.

Slurries will be incubated at room temperature (22^{o}C) for 3 hours, and three 5-mL gas samples will be extracted from the bottle headspaces at 45-minute intervals during the incubation to measure N_2O production over time. Flasks will be continually mixed on a shaker table set at 125 rpm between measurements to equilibrate N_2O between the gas and aqueous phases. Gas samples will be analyzed immediately by gas chromatography by manually injecting each sample directly into a Shimadzu GC-2014 equipped with a 2 m Porapak Q column and a 63Ni electron capture detector. Concentrations are corrected for N_2O solubility in the aqueous phase using the temperature-dependent Bunsen coefficient based on ambient laboratory temperature (Knowles, 1982). The linear rate of N_2O production will be used to determine the rate of denitrification within each flask. Only time periods representing linear production of N_2O are used for calculations due to potential interference of bottle effects (Groffman and Tiedje, 1989). DEA rates are scaled to soil dry-mass (mg-N g-soil^{-1} h^{-1}) to determine the flux of N per unit mass of soil, allowing comparisons across soils of contrasting physical properties.

Microbial biomass N will be measured using the chloroform fumigation incubation method (Jenkinson and Powlson, 1976). Samples are fumigated with chloroform to kill and lyse

microbial cells (releasing cellular N), and fumigated soils are inoculated with fresh 0.2 g soil. All fumigated and unfumigated control samples are incubated at 25°C in the dark for 10 days. During the incubation, microorganisms lysed by chloroform are mineralized to NH_4. Prior to and following incubation, extractable NH_4 and NO_3 will be measured in fumigated and control sediments by incubating soil with 2.0M KCl solution on a shaker table at 125 rpm for one hour to release bound ions into solution. The supernatant is filtered through 2.5 µm Whatman filters using gravimetric filtration. Sediment extracts will be analyzed for NH_4 and NO_3 on a SEAL AQ300 Discrete Analyzer (SEAL Analytical, Inc, Mequon, WI)[2]. Dissolved inorganic N (NH_4 and NO_3) in pre- and post-incubation control soils will be used to calculate net nitrogen mineralization (production of inorganic N) and net nitrification (transformation of NH_4 to NO_3^- via net change in NO_3). We will determine soil moisture using measuring gravimetric water content and drying subsamples at 105°C for 24 hours.

4.1.3. Microbial P Release

The release of P from sediment microbial biomass turnover and cell lysis during hydrologic shifts is a potential source of P to the water column upon rewetting desiccated sediments. The sampled sediments will be analyzed for the P content of the microbial biomass in a similar manner to microbial biomass N described above. Microbial biomass P is calculated from the difference between the amount of inorganic P that is extracted by 0.5M $NaHCO_3$ from fresh soil fumigated with chloroform and unfumigated soil (Brookes et al., 1982).

4.1.4. Exposed Sediment C and N Flux

Dry C and N flux dynamics will be assessed concurrently during the four sampling events around the lake perimeter along transects from the high-water levels to the current lake shoreline, including perennially terrestrial sites to determine natural soil carbon evasion rates (five flux sites total per event. Preliminary data carried out by the Brothers lab has found that carbon dioxide (CO_2) and methane (CH_4) fluxes from the exposed lake bed around Utah Lake can be very high, relative to natural terrestrial fluxes and calculated lake surface fluxes. In addition to the global warming potential of such fluxes, the significant transformation and loss of organic carbon from the sediments to the atmosphere during exposed/dry periods may have consequences on C flux dynamics upon inundation. Measurements will be made using a CO_2/CH_4 gas flux analyzer (Picarro, Brothers Lab) paired with sealed chambers on the sediment surface. A second set of sealed chambers (triplicate) will be installed adjacent to the C collection for manual samples of N_2O production. Ten mL samples will be extracted from the chamber headspace and injected into evacuated vacutainers using a gas-tight luer lock syringe. N_2O observations will be made over a 30-minute sampling window with samples taken at times 0 minutes, 15 minutes, and 30 minutes. Headspace N_2O samples will be transported back to the laboratory and analyzed on the Shimadzu GC-2014 following the same injection methods detailed in section 4.1.1.2. Samples of terrestrial vegetation growing in the seasonally exposed lakebed surface will be collected in 1 m^2 quadrants and analyzed at USU laboratories for dry weight and organic carbon content to assess the potential delivery of fresh and potentially labile

[2] Method detection limits: NH_4 – 0.004 mg N L^{-1}; NO_3 – 0.004 mg N L^{-1}

organic matter to the lake's littoral zone upon inundation. Environmental parameters at each flux site (air and sediment temperature, etc.) will be recorded, and sediment samples will be retrieved for lab analysis of pH and conductivity.

4.2 Sediment Core Sampling Locations and Sampling Schedule

We will collect sediment cores at the same locations as in Task 1. Sediment cores will be taken from four locations (Provo Bay, West shore, Northeast shore, and Southern shore to capture spatial variability at the scale of the lake). We will sample all cores and water used in the experiment during the third week of August (Aug. 16-20; concurrently with Task 1) when the lake levels are at or near the lowest levels of the season, September 6-10; October 18-22, and November 29-December 3 to capture the seasonal heterogeneity and hydrologic fluctuations.

4.3. Sample Transport and Storage

All cores will be transported to the lab on ice. The mechanistic laboratory analyses will be initiated within 24 hours of core extraction and manipulations will be conducted at room temperature 20°C.

4.4. Task Outcomes

The data collected from the mechanistic sediment analyses will provide insight into the processes modulating C, N, and P release, recycling, and removal rates during hydrologic fluctuations in the littoral sediments of Utah Lake.

5. Task 3 – Spatial and Temporal Model on Impact of Drying-Rewetting Sediments

We will work with DWQ's Scott Daly to determine the spatial and temporal extent (duration and frequency) of wetting and drying patterns in littoral areas through Geographic Information System (GIS) analysis and evaluation of daily lake elevation data. This will allow us to develop quantitative relationships for estimating the oxygen demand and nutrient fluxes of re-wetted sediments as a function of the frequency and duration of periods of wetting and drying. These objectives will be completed by DWQ in consultation with the Littoral Sediment Team.

6. Technical Report Preparation

The Littoral Sediment Research Team must complete this scope of work in collaboration with and under the direction of the ULWQS Science Panel. The USU Research Team will:

- Develop the final research work plan in consultation with the Science Panel;
- Be responsive to Science Panel input on the Sampling Analysis Plan, work plan deliverables, results, analysis, final report, and any other interest to the Science Panel; and,
- Make all data and information collected by this contract, or funded by the ULWQS, available to the Science Panel within 45 days of field or laboratory analysis. All data and

information must be made available in electronic format through an electronic file sharing website.

For this task, proposers will compile the methods and results from Tasks 1–3 into one comprehensive report for review by the Science Panel. Proposers should prepare a draft and final report based on feedback from the Science Panel. The report must include the methods, results, and discussion that answer the study objectives.

6.1. Expected Deliverables

- Draft and final technical reports; and
- Final electronic deliverable of raw and interpreted data.

7. Approach for Science Panel Collaboration

The Littoral Sediment Research Team must complete this scope of work in collaboration with and under the direction of the ULWQS Science Panel. The USU Research Team will:

- Develop the final research work plan in consultation with the Science Panel;
- Be responsive to Science Panel input on the Sampling Analysis Plan, work plan deliverables, results, analysis, final report, and any other interest to the Science Panel; and,
- Make all data and information collected by this contract, or funded by the ULWQS, available to the Science Panel within 45 days of field or laboratory analysis. All data and information must be made available in electronic format through an electronic file sharing website.

All team members are committed to openly sharing data with the DWQ and ULWQS in as timely manner as possible. The team will create data spreadsheets, which will be shared online, with the minimum parameters of: analysis date, analyst name, sample identification, concentration of each analyses, calibration information, reagent blanks, check standards

8. Data Management Plan

The Littoral Sediment Team will provide regular monthly progress updates to the DWQ project manager. Progress updates are intended to provide regular communication between the project team and DWQ, coordinate technical assistance, ensure the deliverables and commitments of the contract are satisfied, and to coordinate interactions between to the USU Research Team and the ULWQS Science Panel.

Progress updates will occur by means of:

1. A regular monthly coordination call with the DWQ project manager;

2. Written progress reports to accompany all invoices; and

3. Verbal updates/presentations to the Science Panel at all Science Panel meetings.

Utah State University will provide ongoing oversight of the contract, including financial and technical progress reporting as described in this section.

Written progress reports should include sufficient detail to demonstrate the status of each task, whether deliverables are on track, and if the terms of the grant are being met. Written progress reports may be accomplished in a bulleted summary (not to exceed 2 pages) using the Progress Report Template below (2). Progress reports will include:

- Summary of the work completed for each task and deliverable during the reporting period;
- Summary of work anticipated for each task and deliverable during the next reporting period;
- Estimated completion percentage for each task and deliverable.

Each physical sample collected will be assigned a globally unique identifier before the sample is collected in the field. These identifiers will be affixed to the sampling containers and will accompany the samples in the field and in the laboratory. Data will be immediately entered into the shared google document-excel spreadsheets.

9. Project Milestones and Deliverables

Deliverable due dates are based upon days from the contract award date. The project and all deliverables must be completed with consideration of the milestones in the table below. Any change to the deliverable dates must be mutually agreed upon by DWQ and the selected contractor.

All final products generated by the USU Research Team will be transmitted to DWQ in a mutually agreed upon format prior to the expiration of the contract.

Table 2. The milestones and attending dates for Littoral Sediment Study deliverables to the DWQ and ULWQS

Task	Deliverable	Due Date
Task 2 – Develop Sampling and Analysis Plan	Draft sampling and analysis plan (SAP) Final SAP	July 15, 2021 August 15, 2021
Task 3 – Field (collect and preserve cores) and Laboratory (conduct drying/wetting experiments) Components	Collect and preserve cores for analysis Conduct manipulations and measure C, N and P fluxes	January 1, 2021

Task 4 – Analyze C, N and P data	Electronic datasets and metadata.	January 31, 2021
Task 5–Prepare Technical Report	Draft technical report Final technical report	January 31, 2021 March 31, 2022

11. References

Baldwin, D.S., 1996. Effects of exposure to air and subsequent drying on the phosphate sorption characteristics of sediments from a eutrophic reservoir. Limnol. Oceanogr. 41, 1725–1732. https://doi.org/10.4319/lo.1996.41.8.1725

Baldwin, D.S., Mitchell, A., 2000. The effects of drying and re-flooding on the sediment and soil nutrient dynamics of lowland river–floodplain systems: a synthesis. Regul. Rivers Res. Manag. Int. J. Devoted River Res. Manag. 16, 457–467.

Birch, H., 1960. Nitrification in soils after different periods of dryness. Plant Soil 12, 81–96.

Brookes, P.C., Powlson, D.S., Jenkinson, D.S., 1982. Measurement of microbial biomass phosphorus in soil. Soil Biol. Biochem. 14, 319–329. https://doi.org/10.1016/0038-0717(82)90001-3

Brothers, S., Vadeboncoeur, Y., Sibley, P., 2016. Benthic algae compensate for phytoplankton losses in large aquatic ecosystems. Glob. Change Biol. 22, 3865–3873. https://doi.org/10.1111/gcb.13306

Carlton, R.G., Wetzel, R.G., 1987. Distributions and fates of oxygen in periphyton communities. Can. J. Bot. 65, 1031–1037. https://doi.org/10.1139/b87-143

Goel, R., Carling, G., Li, H., Smithson, S., 2020. Utah Lake sediment–water nutrient interactions. Utah Department of Environmental Quality.

Groffman, P.M., Tiedje, J.M., 1989. Denitrification in north temperate forest soils: relationships between denitrification and environmental factors at the landscape scale. Soil Biol. Biochem. 21, 621–626.

Hupfer, M., Zak, D., Roβberg, R., Herzog, C., Pöthig, R., 2009. Evaluation of a well-established sequential phosphorus fractionation technique for use in calcite-rich lake sediments: identification and prevention of artifacts due to apatite formation. Limnol. Oceanogr. Methods 7, 399–410.

Jassby, A.D., Platt, T., 1976. Mathematical formulation of the relationship between photosynthesis and light for phytoplankton. Limnol. Oceanogr. 21, 540–547.

Jenkinson, D.S., Powlson, D.S., 1976. The effects of biocidal treatments on metabolism in soil—V: A method for measuring soil biomass. Soil Biol. Biochem. 8, 209–213.

Jones, A.S., Aanderud, Z.T., Horsburgh, J.S., Eiriksson, D.P., Dastrup, D., Cox, C., Jones, S.B., Bowling, D.R., Carlisle, J., Carling, G.T., Baker, M.A., 2017. Designing and implementing a network for sensing water quality and hydrology across mountain to urban transitions. JAWRA J. Am. Water Resour. Assoc. 53, 1095–1120. https://doi.org/10.1111/1752-1688.12557

Knowles, R., 1982. Denitrification. Microbiol. Rev. 46, 43.

Koenig, L.E., Baumann, A.J., McDowell, W.H., 2014. Improving automated phosphorus measurements in freshwater: an analytical approach to eliminating silica interference. Limnol. Oceanogr. Methods 12, 223–231.

McComb, A., Qiu, S., 1998. The effects of drying and reflooding on nutrient release from wetland sediments. Environment Australia.
Mitchell, A., Baldwin, D.S., 1998. Effects of desiccation/oxidation on the potential for bacterially mediated P release from sediments. Limnol. Oceanogr. 43, 481–487. https://doi.org/10.4319/lo.1998.43.3.0481
Qiu, S., McComb, A., 1995. Planktonic and microbial contributions to phosphorus release from fresh and air-dried sediments. Mar. Freshw. Res. 46, 1039–1045.
Randall, M.C., Carling, G.T., Dastrup, D.B., Miller, T., Nelson, S.T., Rey, K.A., Hansen, N.C., Bickmore, B.R., Aanderud, Z.T., 2019. Sediment potentially controls in-lake phosphorus cycling and harmful cyanobacteria in shallow, eutrophic Utah Lake. PLOS ONE 14, e0212238. https://doi.org/10.1371/journal.pone.0212238
Scholz, O., Gawne, B., Ebner, B., Ellis, I., 2002. The effects of drying and re-flooding on nutrient availability in ephemeral deflation basin lakes in western New South Wales, Australia. River Res. Appl. 18, 185–196.
Schönbrunner, I.M., Preiner, S., Hein, T., 2012. Impact of drying and re-flooding of sediment on phosphorus dynamics of river-floodplain systems. Sci. Total Environ. 432, 329–337. https://doi.org/10.1016/j.scitotenv.2012.06.025
Shaughnessy, A., Sloan, J., Corcoran, M., Hasenmueller, E., 2019. Sediments in agricultural reservoirs act as sinks and sources for nutrients over various timescales. Water Resour. Res. 55, 5985–6000.
Smith, M.S., Tiedje, J.M., 1979. Phases of denitrification following oxygen depletion in soil. Soil Biol. Biochem. 11, 261–267. https://doi.org/10.1016/0038-0717(79)90071-3
Staehr, P.A., Testa, J.M., Kemp, W.M., Cole, J.J., Sand-Jensen, K., Smith, S. V., 2012. The metabolism of aquatic ecosystems: History, applications, and future challenges. Aquat. Sci. 74, 15–29. https://doi.org/10.1007/s00027-011-0199-2
Vadeboncoeur, Y., Lodge, D.M., 1998. Dissolved inorganic carbon sources for epipelic algal production: sensitivity of primary production estimates to spatial and temporal distribution of 14C. Limnol. Oceanogr. Oceanogr. 43, 1222–1226.
Vadeboncoeur, Y., Peterson, G., Vander Zanden, M.J., Kalff, J., 2008. Benthic algal production across lake size Gradients: Interactions among morphometry, nutrients, and light. Ecology 89, 2542–2552. https://doi.org/10.1890/07-1058.1
Weise, L., Ulrich, A., Moreano, M., Gessler, A., E. Kayler, Z., Steger, K., Zeller, B., Rudolph, K., Knezevic-Jaric, J., Premke, K., 2016. Water level changes affect carbon turnover and microbial community composition in lake sediments. FEMS Microbiol. Ecol. 92, fiw035.